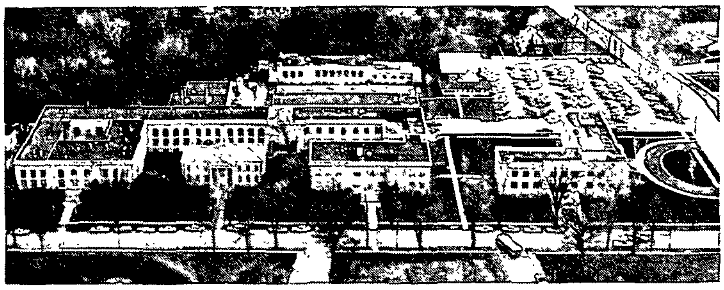

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MORPHOLOGICAL FACTORS IN THE REFINING OF EUCALYPT AND PINUS RADIATA FIBERS

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Morphological Factors in the Refining of Eucalypt and Pinus radiata Fibers

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This manuscript is based on results obtained on a research project by Allender while he was a special student at IPC, and is to be presented at PIRA's Advances in Refining Technologies International Conference in Birmingham, England in December, 1986

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ABSTRACT

Using a solvent exchange, critical-point drying and freeze-fracturing technique, direct evidence of cell wall delamination has been found in *Pinus radiata* and Eucalypt pulps when subjected to refining in a PFI mill. The water retention value (WRV) has been used to measure changes in water uptake in the cell wall with refining. The increases in WRV found for both pulps are mainly affected by fines; however, there is a small but significant change in the WRV for the fines free pulp measured at 3000 g. Furthermore, it appears that apparent density is a sensitive indicator of changes in fiber structure. Limited paper property measurements indicate that the out-of-plane elastic properties of the Eucalypt are superior to those for the *Pinus radiata* and that this measurement is particularly sensitive to fines removal and air drying of the pulp.

1.0 Introduction

The changes in fiber structure produced by refining and their impact on the papermaking process and paper properties have been reviewed by a number of researchers [1,2,3,4]. The major effects are summarized in the table below:

Major Effects of Refining

- Internal fibrillation
- External fibrillation
- Fiber length reduction
- Fines production
- Curls, kinks, microcompressions, straightening

It can be argued that all of these influence interfiber bonding. There is general agreement that internal fibrillation, external fibrillation and fines make a positive contribution to interfiber bond strength, while curl, kinks, and fiber length reduction can have an adverse effect. Clark[2] has debated the relative merits of internal and external fibrillation and believes that the contribution of external fibrillation has been undervalued. In this paper our interest will be mainly focused on internal fibrillation, since it is probably one of the most important effects occurring in the early stages of refining.

Leading proponents of the importance of internal fibrillation include Campbell[5], and Emmerton[6]. Manifestations of the effect include cell wall delamination and increased water uptake. These changes supposedly produce fibers which are more flexible and conformable to one another, and thus lead to improved interfiber bonding, fiber network densification, and improved paper properties. McIntosh[7] and Page and DeGrace[8] reported direct evidence for cell wall delamination and changes in cell wall dimensions. Increased water uptake has long been recognized as a general effect of refining, but the increase due to internal fibrillation alone has not been precisely measured. Techniques for measuring water uptake and changes in cell wall dimensions with refining include the solute exclusion technique,[9] water retention value (WRV),[10,11] and hydrodynamic specific volume[12].

One of the earliest methods of measuring water uptake is the water retention value developed principally by Jayme[10]. Over the course of time the technique has been modified and used to evaluate bleaching, refining, dewatering, and wet pressing. The method is based on the amount of water remaining in a pulp mat after centrifuging. Ellis[11] et al., investigated the change in WRV over a wide range of centrifugal force, and the effects of hardwood versus softwood, refining level and drying. They show that the variation of WRV with log g where g is the acceleration due to gravity generally has two distinct slopes. It is proposed that in the high slope region, water is present both between the fibers in the mat (interfiber water) and within the fibers (intrafiber water). At the "knee" all of the interfiber water is lost, and beyond this point water is removed from within the cell wall with increasing centrifugal force. Scallan and Carles[13] first suggested that an inflection point or plateau would be expected if the pattern of water removal shifted from interfiber to intrafiber with increasing centrifugal force. This idealized interpretation assumes that each layer of the mat is subjected to the same compaction force, which it is not. The effect of fines removal on WRV has been investigated by Thode[14] and Szwarcsztajn and Przybsz[15]. A fines free pulp gives a lower WRV than whole pulp. When using fines-free pulps it might be assumed that the water uptake as measured by the solute exclusion and WRV techniques is due to internal fibrillation only; however, the possible contribution from external fibrillation cannot be ignored.

The above discussion has focused on briefly reviewing a major effect of refining and its measurement, namely internal fibrillation. The results which follow are concerned with the development and measurement of internal fibrillation in a bleached kraft Eucalypt pulp and an unbleached kraft Pinus radiata pulp, which have been refined in a PFI mill. There has been relatively little work reported on the internal fibrillation behavior of hardwoods, and currently Eucalypt is enjoying a lot of attention.

2. Experimental

2.1 Pulp Sources and Treatments

Two commercial never-dried kraft pulps produced at the Australian Paper Manufacturer's Maryvale Mill, Victoria, Australia were used in this study. The bleached Eucalypt was 75% Ash Eucalypt and 25% class D Eucalypt, with a kappa number of 12, and a bleachability of 15. The unbleached Pinus radiata had a kappa number of 30.

The Eucalypt and *Pinus radiata* pulps had consistencies of 28.9% and 25.9%, respectively, on delivery and were beaten with no pretreatment. No preservatives were used. A PFI mill (TAPPI Method T 248 pm-74) was used to beat each pulp. (Eucalypt - 0, 560, 1000, 2000, and 3500 revolutions; Pine - 0, 1000, 3000, 5000, and 7000 revolutions).

After beating, the fines were removed from half of each sample in a Bauer-McNett apparatus using a 200 mesh screen. Small (ca. 50 mL) samples of whole and decrilled pulps were kept in a cold room for later microscopic examination. For all treatment levels two liters of 0.25% consistency pulp was made up and stored in a cold room.

A portion of both the whole and fines free pulps was formed into a mat on coarse filter paper in a Buchner funnel. This was allowed to air dry (ca. 20 g OD).

Pulp Examination and Testing

(a) Canadian Standard Freeness measurements (TAPPI Standard Method T 227m-58) were made on all whole pulp samples.
(b) Water Retention Values (WRV) were measured on whole, decrilled and air dried pulps (dried pulp was soaked for 24 hours before British Standard Disintegrator treatment) after storing 0.25% consistency samples for several days.

The WRV method was modifed from Thode[14] et al. (1960). 19-mm ID Plexiglass tubing was turned to fit a standard 26-mm centrifuge tube. A bronze cap was turned and drilled to fit onto one end of the tube. The cap supported a disk of 100-mesh bronze wire. Grooved plexiglass rings (14 mm high) were inserted into the polypropylene centrifuge tubes to allow room for the accumulation of excess water.

A fiber mat was formed onto the 100-mesh wire in the centrifuge tube from 26 mL of 0.25% consistency pulp. This was accomplished by inserting the centrifuge tube end into a rubber funnel attached to a vacuum flask. The pad was formed under a vacuum of 35 kPa, and a quick acting stopcock was turned off as soon as air could be heard passing through the fiber mat and a drop in vacuum pressure registered on the gage.

The plexiglass tube was immediately stoppered and placed in a centrifuge tube. After centrifuging (25 minutes) the bronze cap was removed and the fiber mat was transferred to a preweighed glass vial using a dissecting needle. The vial was stoppered and weighed on a balance (Mettler AE160). The vial contents were dried unstoppered overnight (100° C), cooled in a desiccator and reweighed. The final OD fiber mat weight was about 0.07 g. Water Retention Values (WRV) were calculated:

$$WRV = \frac{(\text{wet} + \text{vial}) - (\text{dry} + \text{vial})}{(\text{dry} + \text{vial}) - (\text{vial tare})}$$

For each pulp treatment, four replicates of WRV measurements were made at seven different centrifugation levels. For the low range of centrifugal force (70 - 500 g) an IEC model V Centrifuge was used, and for the high range (600 - 42,000 g) a Sorval RC2-B Centrifuge was used.

(c) Fiber length measurements on whole and fines free pulps were made with a Kajaani FS100 Fibre Length Analyser. The weighted average fiber length, the arithmetic mean of fiber lengths in the lower 10% of the fiber population and the percentage fiber fragments (arithmetic) shorter than 0.4 mm were measured (more than 3000 fiber counts per sample). The last two figures provide an indication of the amount of fines present.

(d) Fiber samples of the unbeaten and highly beaten whole pulps and air dried pulps were examined and measured using a Scanning Electron Microscope (SEM). Fibers were taken through an ethanol/water series (5, 20, 50, 70, 85, 95, 100(x3), % ethanol) with at least one hour in each treatment. The samples were CO_2 critical point dried (Ladd Cat. No. 28000), and the fibers were aligned on the edge of clear adhesive tape under a dissecting microscope using fine forceps. The tape was folded over to hold the fibers in position, and then the fibers on the tape were freeze fractured transversely in liquid nitrogen. The tab of fractured fiber ends was glued upright onto a metal stub and coated with gold-palladium (Technics Hummer V) prior to SEM examination.

Fiber dimensions including width, depth, wall thickness and lumen thickness were measured directly from the SEM video screen at magnifications of x1800, x2400 and x3600. At least 30-50 fibers could be counted for each treatment, depending upon the alignment of the fractured fiber ends. Representative photomicrographs were made.

3. Paper Making

Minihandsheets were made in the WRV centrifuge tubes. The forming wire was now 200 mesh bronze (to match standard handsheet forming wires) and the wet mats were made as described in 2(b) above. The minihandsheets were couched using a fitted plastic plunger with a disc of standard laboratory blotter on the end. The bronze cap was removed from the tube and the minihandsheet and blotter were expressed from the tube. The blotter was removed with a dissecting needle.

The wet minihandsheets were arranged on strips of standard blotter and wet pressed and dried for 30 minutes in a laboratory press and dryer combination to minimize shrinkage. The result was a 19-mm diameter minihandsheet having a nominal basis weight of 250 gsm.

4. Paper Testing.

Basis weight and density (IPC soft platen)[16] were measured on six replicate minihandsheets made from whole pulp and selected fines-free and air-dried pulps.

Out-of-plane elastic constant measurements, $C33/\rho$, were made using a 13-mm diameter transducer connected to the IPC laboratory instrument for making such measurements[17]. The disks were then trimmed to 16-mm wide coupons, and the in-plane elastic constant, C_{11}/ρ, measurements were made using the same instrument by carefully standing the coupons on edge. STFI compression strength measurements were then made on the coupons. A trial with commercial medium showed there was no change in measured compressive strength when the sample size was reduced to 16 mm.

Results and Discussion

3.1 Direct observation of cell wall delamination.

Using the fiber preparation techniques described in the experimental section, typical SEM's of sectioned *Pinus radiata* and Eucalypt fibers are shown in Figures 1, 2, 3, and 4, at different levels of refining. Figures 2 and 4 show that delamination is a feature of the secondary wall of refined *Pinus radiata* and Eucalypt fibers, respectively. This appears to be the first time this evidence has been reported for these species. In the case of Pinus radiata, Kibblewhite[18] was unable to show secondary cell wall delamination using a freeze-fracture technique. The present demonstration of delamination is attributed to the improved preparation technique of gradual alcohol infiltration, critical point drying followed by freeze-fracturing. No mention of Eucalypt or other hardwood cell wall delamination has been found in the literature.

Figure 1. Unrefined latewood fiber Pinus radiata, x 1800.

The number of delaminations was similar in both pulps and varied between two and four major separations and often many other minor layers (Figures 2-4). This agrees with the observations of Page and deGrace[8]. On theoretical grounds, any delamination would greatly improve fiber flexibility (Emerton[6] p. 98).

It was apparent from examining fibers at different refining levels that delamination was a heterogeneous process. In unbeaten Pinus radiata and Eucalypt samples it is estimated that up to about 20% of the fibers examined showed some secondary wall delamination, while at the highest levels of refining about 95% did. The lightly refined Pinus radiata latewood fibers showed more evidence of delamination than the earlywood fibers.

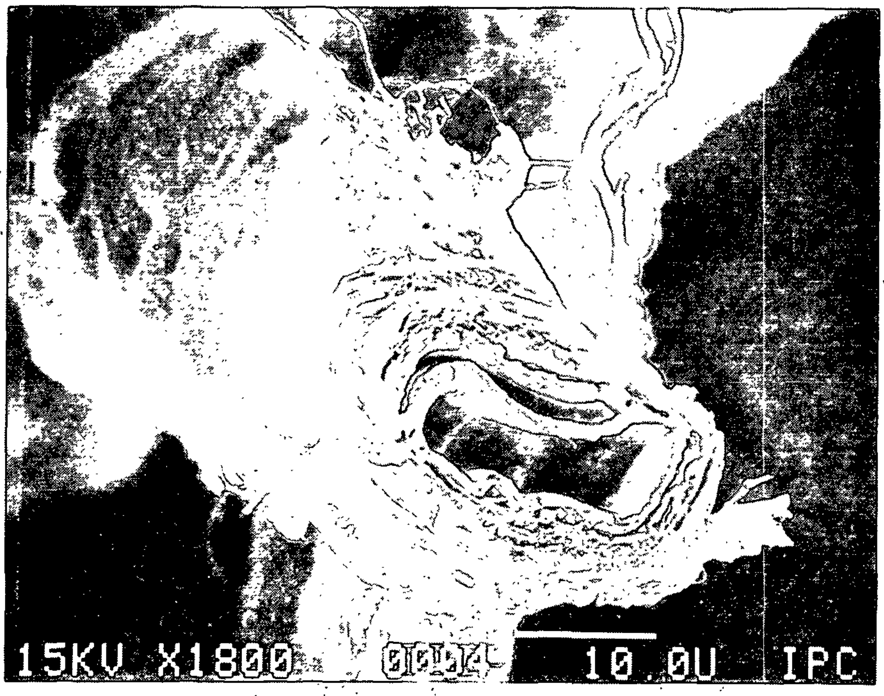

Figure 2. Refined 7000 revolutions PFI latewood Pinus radiata, x 1800.

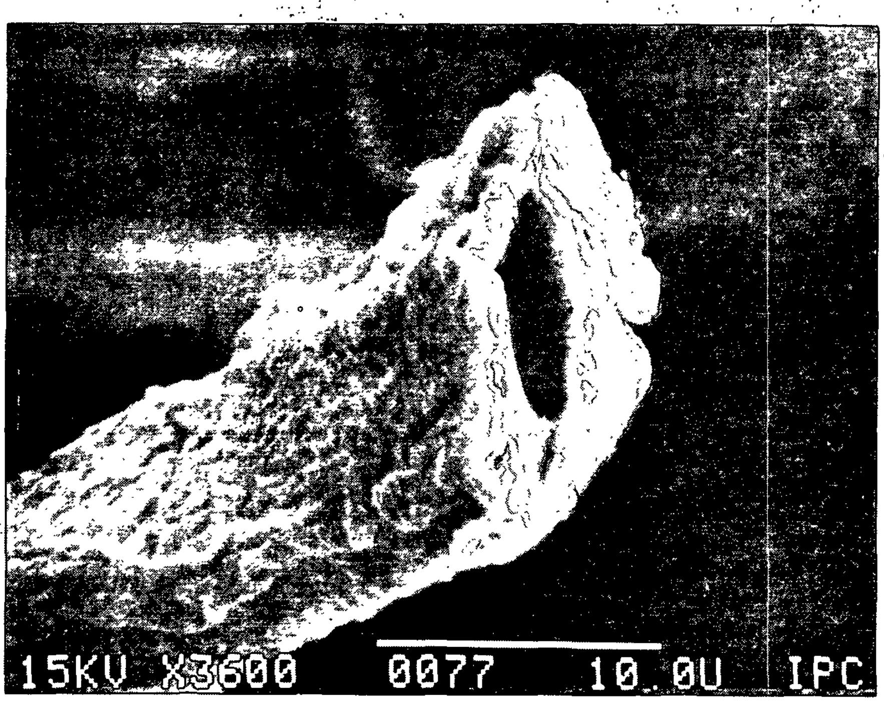

Figure 3. Unrefined fiber Eucalypt, x 3600.

Fiber length and cell wall dimension changes

The weighted average fiber lengths given in Table 1 show effectively no change with refining for the Eucalypt and a slight reduction for the Pinus radiata. Fines production for both pulps was increased with refining but were substantially reduced by the 200 mesh screen (see fines free results Table 1.)

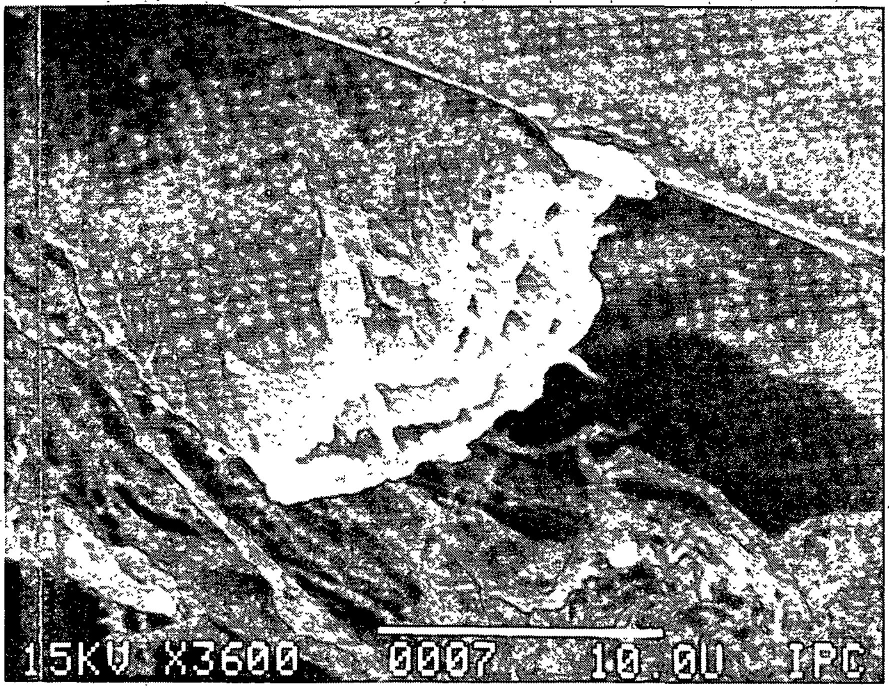

Figure 4. Refined 3500 revolutions PFI Eucalypt, x 3600.

Table 1. Pinus Radiata and Eucalypt Fiber Lengths and Fines

Pulp	PFI Revs	Weighted Fiber Length, mm	Fiber Length Lower 10%, mm	% < 0.4 mm
EUCALYPT (Whole Pulp)	0	0.66	0.13	36.3
	560	0.69	0.11	37.7
	1000	0.69	0.12	36.5
	2000	0.69	0.10	39.7
	3500	0.68	0.09	42.5
EUCALYPT (Fines Free Pulp)	0	0.72	0.32	18.0
	560	0.71	0.26	24.6
	1000	0.71	0.25	24.5
	2000	0.72	0.25	25.0
	3500	0.73	0.27	22.9
PINUS RADIATA (Whole Pulp)	0	1.96	0	46.3
	1000	1.91	0	49.5
	3000	1.90	0	48.7
	5000	1.85	0	50.5
	7000	1.81	0	54.5
PINUS RADIATA (Fines Free Pulp)	0	2.08	0.22	19.8
	1000	2.03	0.29	15.5
	3000	2.07	0.29	15.8
	5000	1.98	0.26	18.5
	7000	1.88	0.23	21.8

Cell wall dimensions are given in Table 2. Unfortunately, the fiber population examined is not large enough to draw statistically significant conclusions. Nevertheless, we shall discuss some of the trends that were observed.

Table 2. Mean fiber dimensions of unbeaten and beaten freeze fractured Pinus radiata and Eucalypt fiber in the swollen state.

PINUS RADIATA	Fiber Width (μm)	Fiber Thickness (μm)	Cell wall Thickness (μm)	Lumen Thickness (μm)
Earlywood				
unbeaten 0 revs.	33.1	16.2	3.5	9.2
unbeaten 600 g	----	----	2.8	7.9
unbeaten 41,000 g	----	----	2.4	5.7
unbeaten air-dried	----	----	2.8	9.3
beaten 7000 revs.	42.3	17.0	6.0	6.1
beaten 600 g	----	----	4.8	6.1
beaten 41,000 g	----	----	3.6	3.2
beaten air-dried	----	----	2.2	2.7
Latewood				
unbeaten 0 revs.	38.0	14.0	3.2	8.3
beaten 7000 revs.	26.5	18.0	5.6	8.9
EUCALYPT				
unbeaten 0 revs.	13.0	6.9	1.7	1.9
unbeaten 600 g	----	----	2.1	3.1
unbeaten 41,000 g	----	----	2.0	3.1
beaten 3,500 revs.	13.9	7.8	2.8	2.1

The Pinus radiata earlywood fibers showed signs of collapse (i.e., their width increased) while the latewood fibers tended to become more circular (i.e., their width decreased while their thicknesses increased) with refining. The changes in the Eucalypt were smaller and showed a slight increase in width and thickness dimensions. Both pulps showed a significant increase in cell wall thickness with refining, i.e, 71% earlywood Pinus radiata, 75% latewood Pinus radiata, and 65% Eucalypt. Centrifuging and air drying reduced the cell wall thickness of the earlywood Pinus radiata fibers, the reduction being greatest for the refined fibers. In contrast centrifuging produced a slight increase in cell wall thickness of the Eucalypt (only unrefined Eucalypt fibers were examined).

The lumen dimensional changes generally follow the cell wall thickness changes. Evidence of fiber collapse (i.e., decrease in lumen thickness) is more prevalent for the earlywood Pinus radiata fibers; refining, centrifuging and air drying all result in a decrease in lumen height. The latewood Pinus radiata and Eucalypt fibers, despite the paucity of data, do not show evidence of collapse, and if anything an increase in lumen height is found.

3.2 Water Retention Values.

The variation of WRV with centrifugal force is shown in Figures 5 and 7 for the whole pulps of Pinus radiata and Eucalypt at three PFI refining levels. The separation of the curves with refining in the low slope region is not as pronounced for the Eucalypt as it is for the Pinus radiata pulp. The effect of fines removal on WRV's is shown in Figures 6 and 8. The curves for each refining level tend to collapse onto a common curve, particularly for the Eucalypt pulp in both the high and the low slope regions. However, as we shall see shortly, there are still significant changes in WRV as a function of refining in the low slope region. In the high slope region, water is centrifuged out from the network, and since the network is not incompressible, its compaction will also aid in further water removal. The rate of water loss in the high slope region is greater for the Eucalypt and is relatively insensitive to refining. The Pinus radiata does show an increase in water removal rate with refining, although it is still less than the Eucalypt at the highest level of refining. Mat compressibility behavior is presumably an important aspect of WRV. In this situation, however, the mat is not uniformly densified. As the mat is densified the free water will be readily removed, and finally a steady state is more or less arrived at where the centrifugal force will no longer remove water from certain sizes of capillary. The model of fiber mat compaction proposed by Osaki[19] et al., suggests that mat compaction in the high slope region could result from an increase in the number of fiber contacts, whereas the low slope region is controlled by fiber cell wall compaction. Fiber compaction will be dependent on fiber structure and composition.

Examining in more detail the effects of refining, fines removal, and drying on changes in the cell wall, the variation of WRV at 3000 g with PFI revolutions is shown in Figures 9 and 10. A small but significant increase in WRV of 9.1% and 5.4% for the Pinus radiata and Eucalypt pulps with fines removed is found over the refining range used for each pulp. Air drying results in a 31% and 33% loss in the unrefined WRV values for the Pinus radiata and Eucalypt pulps, respectively.

Furthermore, since there is no significant change in water retention value with refining when the pulps are air dried, then one effect of internal fibrillation (i.e., increase in WRV) is lost upon drying. This does not necessarily imply that related refining effects are lost upon drying, i.e., fiber flexibility, fiber collapse.

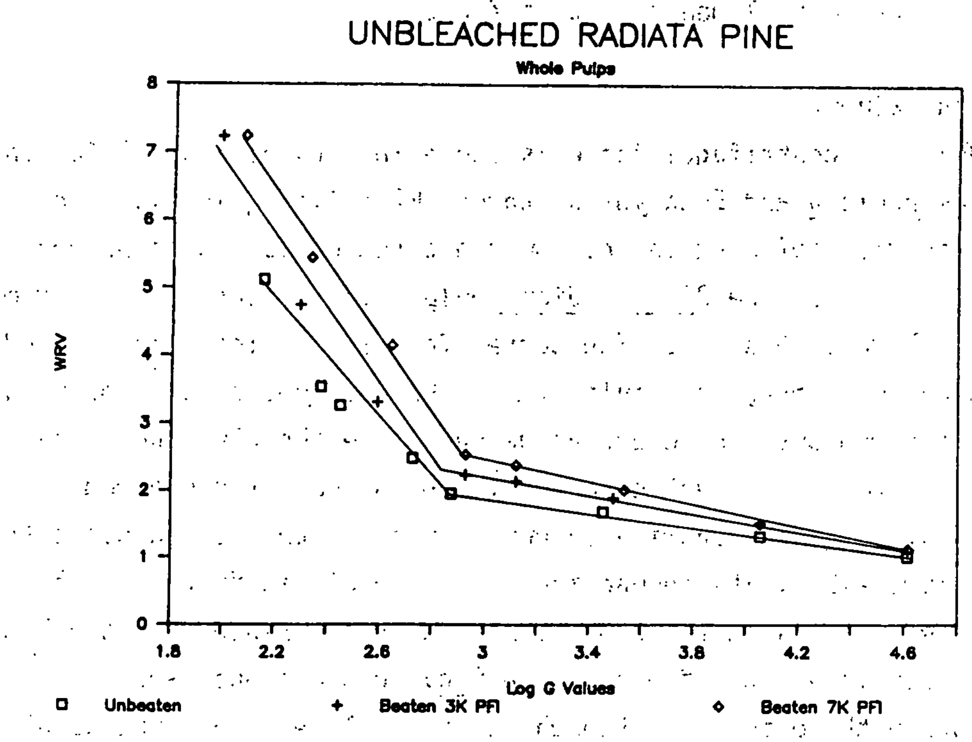

Figure 5. Variation of water retention values with centrifugal force.

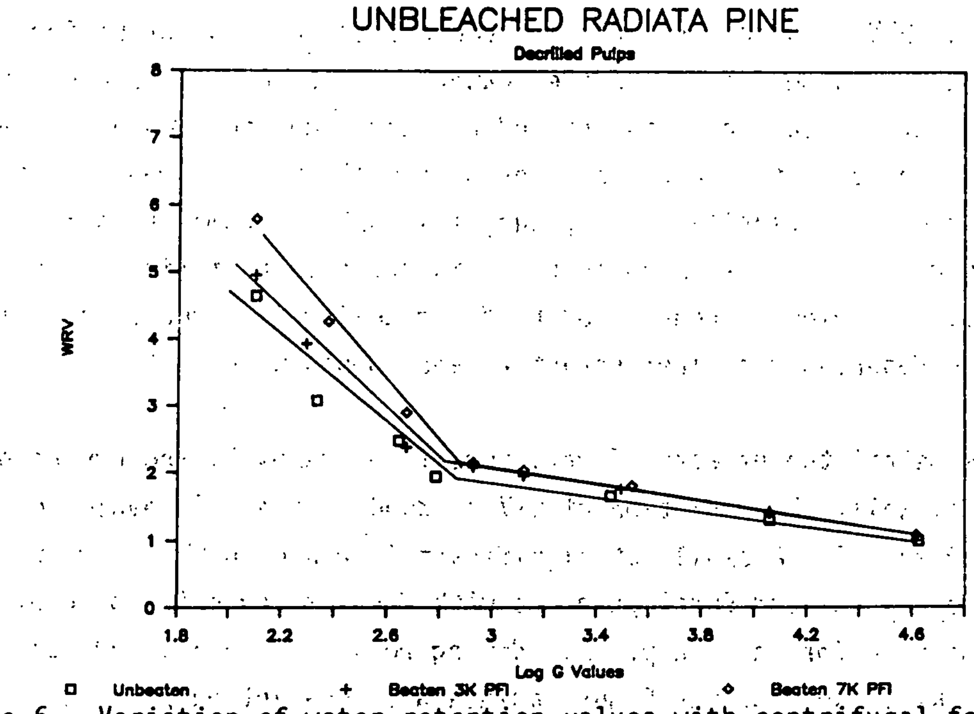

Figure 6. Variation of water retention values with centrifugal force.

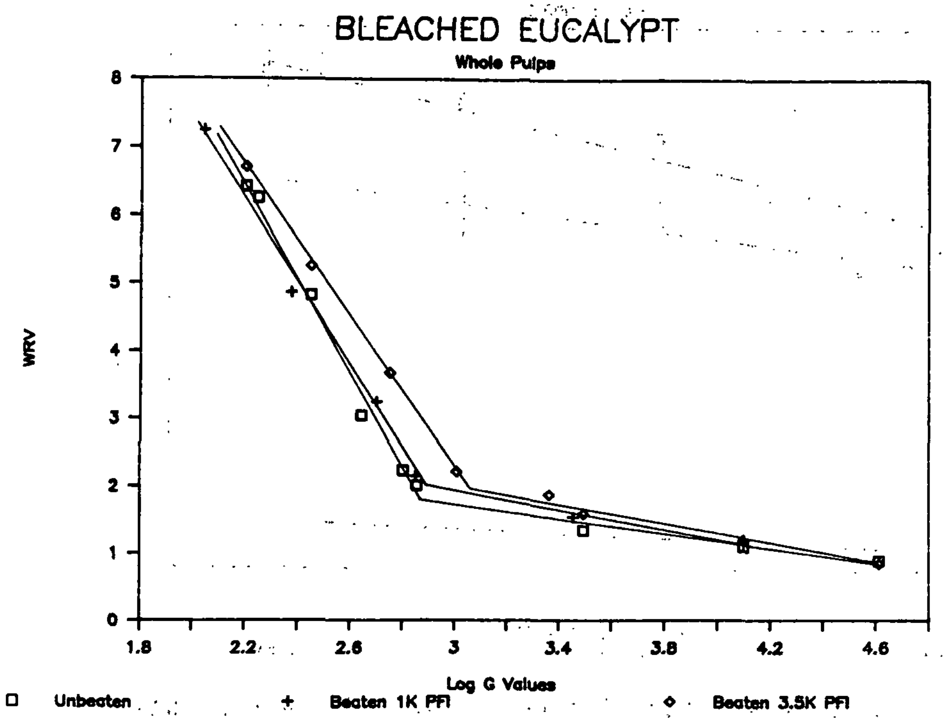

Figure 7. Variation of water retention values with centrifugal force.

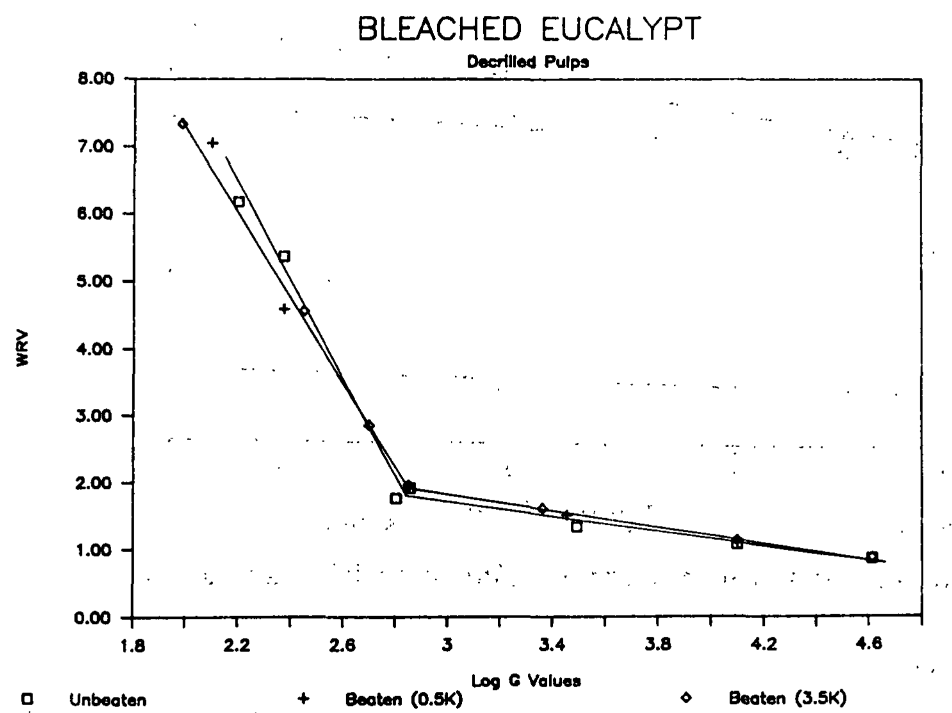

Figure 8. Variation of water retention values with centrifugal force.

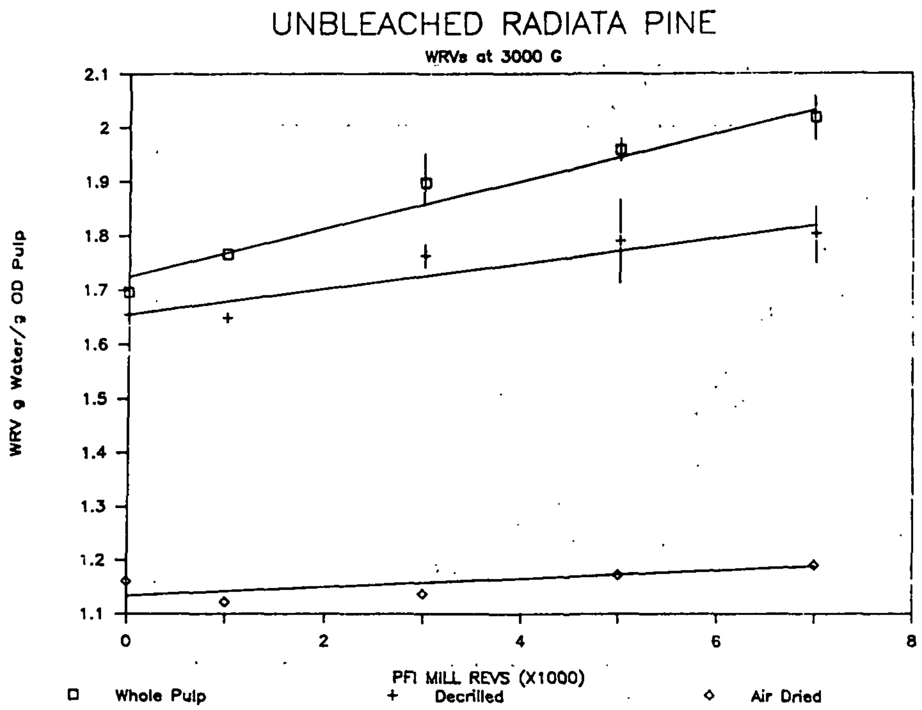

Figure 9. Variation of water retention value with PFI revolutions.

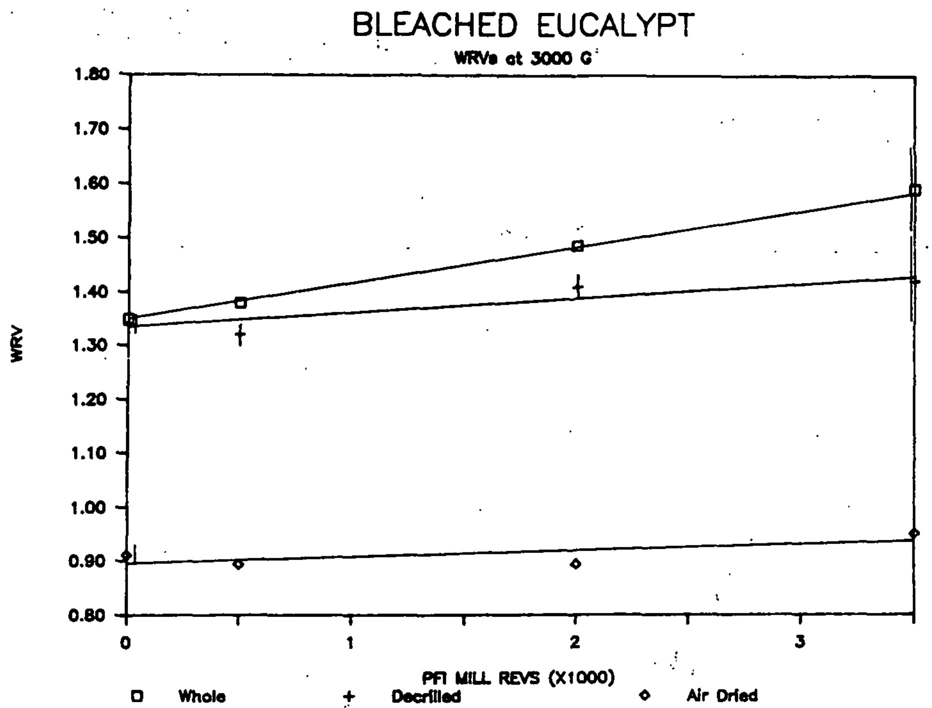

Figure 10. Variation of water retention value with PFI revolutions.

3.3 Paper Properties

Limited paper property measurements were made on minihandsheets (19-mm dia.) having a nominal basis weight of 250 g/m^2. These included basis weight, caliper, ultrasonic elastic constants and compressive strength measurements.

The results are summarized in Table 3 together with WRV's at 3000 g. The variation of apparent density with WRV is shown in Figure 11 for handsheets made from whole pulp, fines-free and air-dried whole pulps. The dominant effect of fines is again seen when we compare the whole and fines free pulp curves. At the highest refining level, removal of fines produces a large reduction in WRV, and only a small drop in apparent density for the Pinus radiata. Essentially the same changes are seen for the Eucalypt except that the change in apparent density is negligible. The overall change in apparent density of the fines-free handsheets is 23.4% and 24.2% for the Pinus radiata and Eucalypt pulps, respectively, which correspond to changes of 9.1% and 5.4% in WRV, as noted above. We might therefore conclude that apparent density is a more sensitive indicator of changes in cell wall structure (internal fibrillation). Air drying, as we have already seen, produces a large overall reduction in WRV. It is believed that drying effectively negates the effect of fines and internal fibrillation, as well as reducing the amount of intrafiber water present, i.e., the changes in WRV with refining are not significant at least for the two pulps examined here. Nevertheless, the effects of refining on sheet densification are still evident, since the change in density from unrefined to the highest level of refining is 17% and 10% for the Pinus radiata and Eucalypt, respectively.

It has been found that many strength related properties correlate with apparent density. Furthermore, a reasonable correlation between relative bonded area and apparent density has been demonstrated by Waterhouse[20], which for a given pulp, is independent of the level of wet pressing and refining. Therefore, to a first approximation, apparent density may be used as a measure of relative bonded area.

The variation of in-plane and out-of-plane specific elastic constants with apparent density (produced by refining) are shown in Figures 12 and 13. At a given density level the Pinus radiata gives a slightly higher in-plane elastic constant; however, the out-of-plane elastic constant of the Eucalypt is significantly higher than the Pinus radiata. A good correlation has also been found by Baum[21] between out-of-plane strength, e.g., ZDT, and out-of-plane specific modulus, Therefore the Eucalypt would be expected to have superior out-of-plane strength when compared with the Pinus radiata at a given apparent density.

The changes in properties with fines removal and air drying are summarized in Table 4 for two levels of refining. The greatest property change experienced by both pulps when fines are removed is a reduction in the out-of-plane elastic constant. The changes in in-plane constants are much smaller by comparison. This result coincides with the expectation that the out-of-plane elastic constant will be more sensitive to interfiber bonding, and thus fines level, than the in-plane elastic constant. As already noted the largest change in WRV is at the highest level of refining for both pulp types.

Table 3. WRV and minihandsheet properties.

PFI Revs.	CSF	WRV 3000 g	Basis Wt. g/m^2	Density g/cc	$\overline{C}/\rho$ $(km/sec)^2$	C_{33}/ρ $(km/sec)^2$	STFI $\sigma c/\rho$ Nm/g
Bleached Kraft Eucalypt							
0	612	1.347	260	0.808	9.14	0.097	24.9
500	555	1.376	258	0.840	10.44	0.193	31.6
1000	520	1.537	253	0.886	11.34	0.231	35.3
2000	460	1.486	248	0.889	11.78	0.280	36.1
3000	345	1.586	271	0.923	12.13	0.349	37.4
Fines Free							
0	---	1.342	207	0.744	8.80	0.0729	23.7
3500	---	1.415	221	0.924	12.78	0.235	36.5
Air Dried							
500	---	0.893	223	0.737	7.65	0.0655	18.4
2000	---	0.894	223	0.773	7.92	0.0751	21.1
Unbleached Kraft <u>Pinus radiata</u>							
0	737	1.696	212	0.849	10.63	0.068	31.0
1000	694	1.766	250	0.908	12.43	0.144	35.4
3000	610	1.897	246	0.941	13.78	0.181	42.6
5000	526	1.958	268	0.957	14.17	0.181	42.9
7000	420	2.018	243	0.994	14.38	0.189	43.9
Fines Free							
0	---	1.655	228	0.788	9.57	0.044	28.7
7000	---	1.804	212	0.972	13.21	0.123	43.1
Air Dried							
0	---	1.160	255	0.695	6.99	0.018	17.8
7000	---	1.190	230	0.869	9.43	0.106	29.1

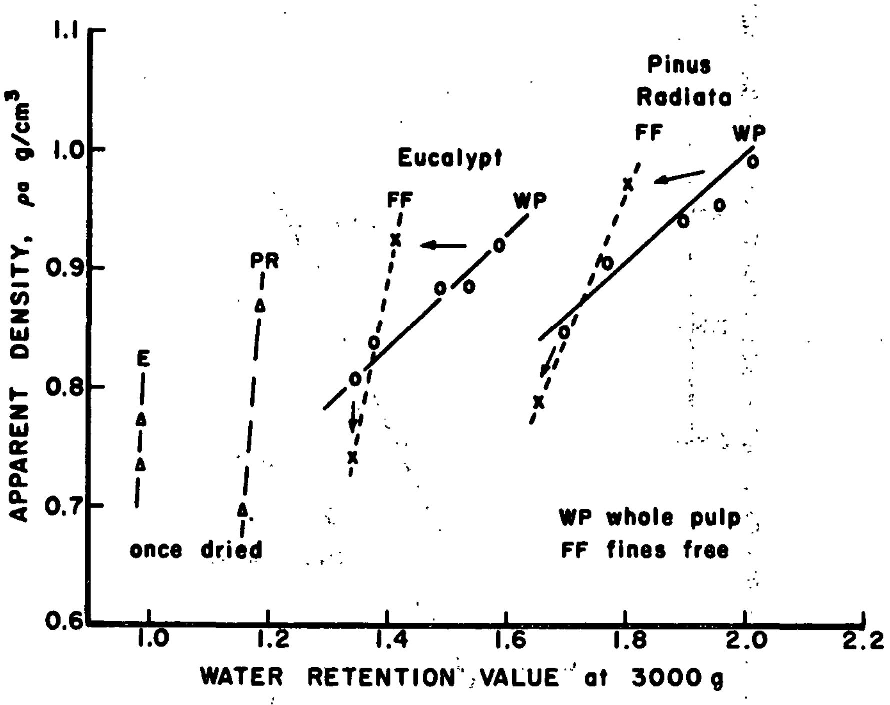

Figure 11. Variation of sheet density with water retention value.

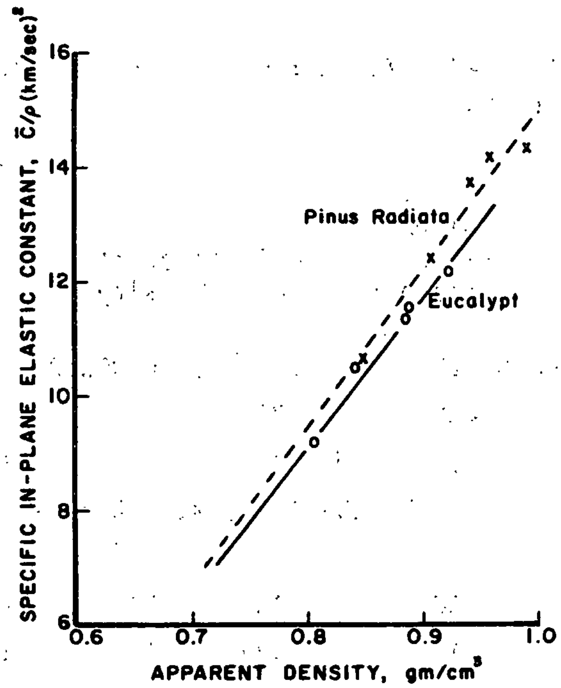

Figure 12. Variation of elastic constants with apparent density.

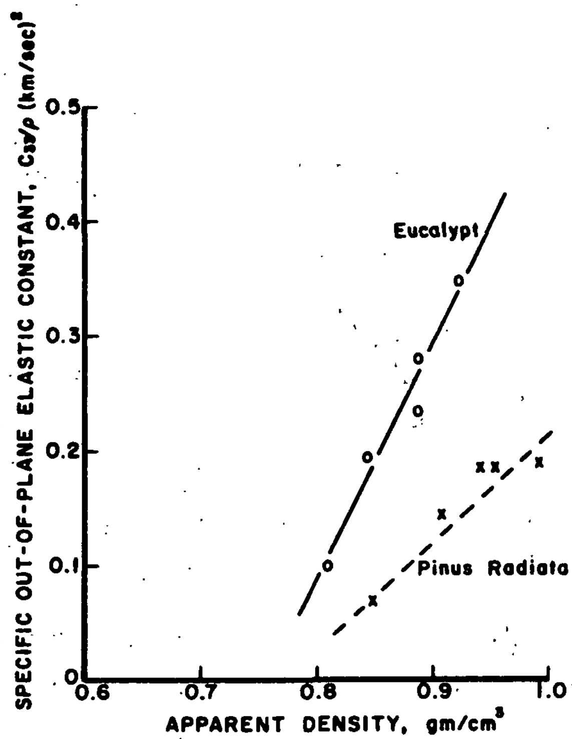

Figure 13. Variation of elastic constants with apparent density.

Table 4. Changes in Properties with fines removal and air drying relative to whole pulps.

EUCALYPT					PINUS RADIATA				
CSF mL	Apparent Density %	WRV %	In-plane Elastic Constant %	Out-of-plane Elastic Constant %	CSF mL	Apparent Density %	WRV %	In-plane Elastic Constant %	Out-of-plane Elastic Constant %
CHANGE IN PROPERTIES WITH FINES REMOVAL									
612	-8	0	-4	-32	737	-7	- 2	-11	-34
345	0	-11	+5	-33	420	-2	-11	- 7	-37
CHANGE IN PROPERTIES WITH AIR DRYING									
EUCALYPT					PINUS RADIATA				
555	-12	-35	-27	-66	737	-24	-32	-34	-74
460	-13	-39	-33	-73	420	-13	-41	-34	-44

Air drying produces large reductions in all of the properties measured, with the largest change again occurring in the out-of-plane elastic constant. The reduction in both in-plane and out-of-plane elastic constants is presumed to be due, in the former case, to a reduction in both interfiber bonding and fiber modulus, and in the latter case, to a reduction in interfiber bonding.

The variation of STFI compressive strength with densification (by refining) for the Pinus radiata and Eucalypt pulps is shown in Figure 14. The performance of the two pulps is almost identical, and it is noted that the fines-free results do not deviate significantly from the whole pulp correlation (based on the data of both pulps).

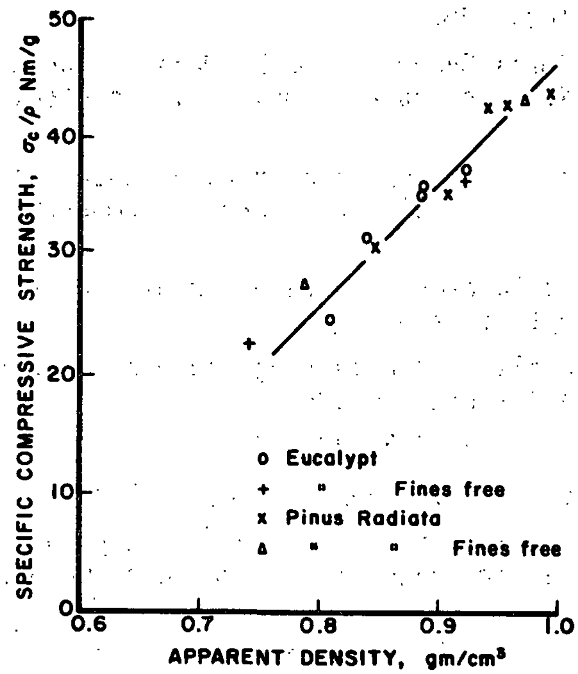

Figure 14. Variation of specific compressive strength with apparent density.

Conclusions

Using a solvent exchange, critical-point drying and freeze-fracturing technique, direct evidence of cell wall delamination has been found in Pinus radiata and Eucalypt pulps when subjected to refining in a PFI mill. The water retention value (WRV) has been used to measure changes in water uptake in the cell wall with refining. The increases in WRV found for both pulps are mainly affected by fines; however, there is a small but significant change in the WRV for the fines free pulp measured at 3000 g. Furthermore, it appears that apparent density is a sensitive indicator of changes in fiber structure. Limited paper property measurements indicate that the out-of-plane elastic properties of the Eucalypt are superior to those for the Pinus radiata and that this measurement is particularly sensitive to fines removal and air drying of the pulp.

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